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I also certify that the attached copy of the request for grant of a Patent (Form 1/77) bears a correction, effected by this office, following a request by the applicant and agreed to by the Comptroller-General.

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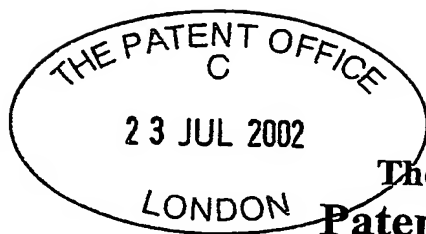
## PRIORITY DOCUMENT

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*Andrew Garsy*

Dated 16 July 2003



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P01/7700 0.00-0217078.5

Patents Form 1/77

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The  
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1/77

### Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

The Patent Office  
Cardiff Road  
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1. Your reference

RFW/JD/P33084

2. Patent application number

(The Patent Office will fill in his part)

0217078.5

23 JUL 2002

3. Full name, address and postcode of the or of each applicant (underline all surnames)

Patents ADP number (*if you know it*)

If the applicant is a corporate body, give the country/state of its incorporation

~~SmithKline Beecham p.l.c.~~

~~980 Great West Road, Brentford, Middlesex TW8~~

~~9GS, Great Britain~~

GLAXO GROUP LIMITED  
GLAXO WELLCOME HOUSE  
BERKELEY AVENUE  
GREENFORD, MIDDLESEX

UB6 0NN

4. Title of the invention

Formulation

5. Name of your agent (*if you have one*)

Corporate Intellectual Property

"Address for service" in the United Kingdom to which all correspondence should be sent  
(including the postcode)

GlaxoSmithKline  
Corporate Intellectual Property CN925.1  
980 Great West Road  
BRENTFORD  
Middlesex TW8 9GS

Patents ADP number (*if you know it*)

7960982003

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or each of these earlier applications and (*if you know it*) the or each application number

Country      Priority application number      Date of filing  
(*if you know it*)      (*day / month / year*)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application      Date of filing  
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8. Is a statement of inventorship and of right

**Patents Form 1/77**

9. Enter the number of sheets for any of the following items you are filing with this form.  
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Continuation sheets of this form  
Description  
Claim(s)  
Abstract  
Drawings

5

*RM*

10. If you are also filing any of the following, state how many against each item.

Priority Documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*)

Request for substantive examination (*Patents Form 10/77*)

Any other documents  
(please specify)

11.

We request the grant of a patent on the basis of this application

Signature

*R F Walker*

Date 23-Jul-02

R F Walker

12. Name and daytime telephone number of person to contact in the United Kingdom

R F Walker 020 80474485

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## Formulation

This invention relates to dentrifice formulations, in particular to a dentrifice formulation which can be stored in a pressurised container from which it can be dispensed as a foam.

5       Dentrifices are commonly provided as a paste, i.e. a toothpaste, in a collapsible container from which they can be extruded. It is also known to provide a dentrifice formulation as a foam, e.g. as disclosed in DE-A-100 08 837. Such formulations generally comprise a fluid mixture containing one or more abrasive, thickener, flavour etc. together with a propellant, normally a liquefied gas with a  
10       boiling point below ambient temperature to drive the formulation out of its container and to expand to foam the formulation.

It is an object of the present invention to provide a foaming dentrifice formulation with improved properties.

According to this invention a dentrifice formulation is provided comprising a  
15       fluid mixture which includes a liquefied gas propellant and containing a particulate abrasive, characterised in that the particulate abrasive comprises 1-10% by weight of the mixture, has a particle size in the range 5-40 microns and comprises a combination of a more hard and a less hard abrasive.

The formulation is normally stored in a container provided with a release  
20       valve, under a pressure corresponding to the vapour pressure of the liquefied propellant at the storage temperature, and on opening the valve the formulation is expelled as a foam, e.g. onto a toothbrush head.

The formulation preferably also contains one or more of the following.

One or more humectant, typically in a proportion of 25-75 wt%, preferably  
25       45-55 wt%, especially  $50 \pm 2$  wt%. Humectants are added to protect the formulation from drying out and to provide consistency and protection against cold. Suitable humectants include sorbitol and glycerol. Suitably a mixture of sorbitol and glycerol may be used e.g. in a sorbitol:glycerol ratio in the range 1:1.5 - 1.5:1. Other  
30       humectants may be used including xylitol, mannitol, 1,2-propylene glycol or mixtures of these polyols.

One or more slurring/suspending agent, typically in a proportion of 1-5 wt%, preferably 2-3 wt%. A preferred slurring agent is polyethylene glycol, e.g. of molecular weight in the range 200 – 800, typically ca. 300.

5 One or more foaming agent. Typically a surfactant may be used as a foaming agent. Suitable surfactants include anionic surfactants such as a sodium alkyl sulphate with a 12-18 carbon atoms in the alkyl chain, such as sodium lauryl sulphate. Zwitterionic, ampholytic and non-ionic surfactants may also be used. A mixture of surfactants may be used. Suitably the surfactant may comprise 0.1-3.0 wt% of the formulation, preferably 1- wt%.

10 One or thickening agent. A preferred thickening agent is a xanthan gum. Typically the thickening agent may comprise 0.1-1.0 wt% of the formulation, typically 0.2-0.5 wt%. It is found that use of a xanthan gum can lead to a more creamy foam with improved flow and texture characteristics.

15 One or more pH regulator, preferably to maintain the pH at 7.5-8.5, especially at ca. pH 8.0. Such a pH is found suitable to avoid corrosion of the aluminum containers which are commonly used for containing such formulations. A suitable pH regulator is sodium hydroxide.

One or more other excipient such as a sweetener, colour, preservative, flavours etc., typically comprising up to ca. 2 wt% of the formulation.

20 One or more active material such as an antimicrobial agent, tartar inhibitor, remineralisation agent, vitamin, fluoride, e.g. sodium fluoride, typically comprising up to ca. 0.5 wt% of the formulation.

25 Many other examples of materials of these types are known in the state of the art, e.g. in DE-A-100 08 837, the content of which is incorporated herein by way of example only.

30 Preferably the formulation comprises 9 wt% or less, e.g. 3-7 wt% abrasive, especially 4.5-6 wt%, typically ca. 5 wt%. Preferably the particle size of the abrasive is 30 microns or less, preferably 10 microns or less. A mixture of at least one less hard and at least one more hard particulate abrasive is used, typically in a proportion more hard : less hard in the range 1 : 1-5, suitably in the range 1 : 2.5 – 3.5. Suitably the abrasive material may be a silica. Suitable silicas include those known as Zeodent 124™ and Zeodent 623™.

A suitable propellant is a liquefied gas that generates a pressure of ca. 30 psi (ca. 2 kg/cm<sup>2</sup>). Many propellants are known which can achieve this, suitably a commercial product "Butane 30" comprising a mixture of n-butane, i-propane and n-propane. Typically the formulation may comprise ca. 3 wt% of such a liquefied gas.

The remainder of the formulation may comprise water, typically comprising ca. 25-40 wt%, preferably 30-40 wt% of the formulation.

A typical formulation according to this invention therefore comprises: one or more humectant 45-55 wt%, slurring agent 2-3 wt%, foaming agent 1-2 wt%, abrasives 3-7 wt%, preferably 3-5 wt %, thickening agent 0.2-0.5 wt%, flavour, active and sweetener 0-2 wt%, pH adjuster if necessary to provide pH of 8.5 +/- 0.2, water 30-40 wt % preferably 35 +/- 1 wt%. This fluid formulation is preferably charged into a metal container with a dispensing valve, at a proportion of 97 wt% with 3 wt% propellant providing a pressure of 30 psi (ca. 2 kg/cm<sup>2</sup>). such as the above-mentioned Butane 30.

The proportion and particle size of the abrasive are found to optimise the combination of suitability for flow of the formulation out through the valve and effective tooth cleaning. The preferred materials and their proportions described above also contribute to improved flow and handling of the formulation.

A typical process for making the formulation of this invention may involve the steps of:

1. Adding a suitable quantity of water to a mixing vessel.
2. Adding sweetener and active to the water and agitating until dissolved or suspended.
3. Adding the humectant and agitating until homogeneous.
4. Sieving the abrasive to break up any lumps. A 500 micron sieve is generally suitable.
5. Slowly adding the abrasive to the mixture while mixing.
6. Slurring the thickening agent and slurring agent and add to the mixture, agitate until homogeneous.
7. Mixing, optionally transferring to a mixer.

8. Slurrying the flavour and foaming agent and adding to the mixture, mixing until homogeneous.

9. Adjusting the pH.

10. Mixing until homogeneous.

5 This fluid mixture may then be charged into suitable valued containers together with a suitable quantity of propellant.

The formulation may be used in a generally conventional manner involving opening the valve of the container to allow the internal pressure to expel the formulation onto a toothbrush. The invention also provides a valved container  
10 containing a formulation as described above.

The invention will now be described by way of example only.

A dentrifice formulation was prepared having the following composition:

	Function	Component	wt%	g per 500g
	Humectant	Sorbitol 70% non-crystallizing	28.000	140
15	Humectant	Glycerin	22.00	110
	Suspending agent	PEG 6	2.500	12.5
	Foaming agent	Empicol 0303 30% solution	5.000	25.000
	Sweetener	Sodium saccharin	0.300	1.500
	Active	Sodium fluoride	0.306	1.530
20	Flavour	Flavour C180 (minty)	1.000	5.000
	Abrasive (hard)	Zeodent 124	1.330	18.350
	Abrasive (soft)	Zeodent 623	3.670	18.350
	Thickener	Xanthan (Keltrol F)	0.250	1.250
	pH adjuster	35% NaOH solution	0.250	1.250
25	<u>Water</u>		<u>35.394</u>	<u>176.97</u>
	Total		100.00	500

These proportions could be varied by +/- 10%. This fluid formulation was made by a process as described above, involving

1. Adding a suitable quantity of water to a mixing vessel.

30 2. Adding sweetener and active to the water, mixing until dissolved using a circular paddle stirrer on a Heidolph.

3. Adding the glycerol and sorbitol to the batch, mixing until dissolved using a circular paddle stirrer on the Heidolph.
4. Sieving the abrasive to break up any lumps. A 500 micron sieve is generally suitable.
- 5 5. Slowly adding the abrasive to the mixture, mixing using a circular paddle stirrer on the Heidolph..
6. Slurrying the thickening agent and slurrying agent and add to the mixture, agitate until homogeneous.
7. Transferring to an Ultra Turrux mixer and mixing for 5 minutes.
- 10 8. Slurrying the flavour and foaming agent and adding to the mixture, mixing until homogeneous with a circular paddle stirrer on the Heidolph.
9. Adjusting the pH to pH 8 (+/- 0.5 using the NaOH.
10. Mixing until homogeneous using circular paddle stirrer on the Heidolph.
- 15 This fluid mixture was charged into valved containers together with Butane 30 propellant in a 97:3 ww ratio.